**Supporting Information**

**The effective adsorption of Ni(II) and nitrate from aquatic systems by superparamagnetic MoS2/γ-Fe2O3 nanocomposites: optimization through RSM-CCD design**

*Somayeh Ostovar,a \* Hamideh Saravani, a\* Maryam Akbari a Amanolah Salehpour, a* [*Mohammad Sabaghi*](mailto:mohammadsabaghi8877@gmail.com)*,a Esmaiel Rezazadeh a*

a Department of Chemistry, University of Sistan and Baluchestan, Faculty of Sciences, PO Box98135-674, Zahedan, Iran.

\*S.O. somayeh.ostovar.s@gmail.com, \*H.S. [saravani@chem.usb.ac.ir](mailto:saravani@chem.usb.ac.ir)

**1-Experimental.**

**1.1. Materials and reagents**

The starting material of molybdenum disulfide (MoS2), including (NH4)6Mo7O24.4H2O (≥99.0%), and H2NCSNH2 (≥99.0%), and γ-Fe2O3 nanoparticles, including FeCl3.6H2O (≥98.0%), FeCl2.4H2O (≥99.0%), HCl (35–38%), NH4OH (25%), tetramethylammonium hydroxide ([25 wt.% in H2O)](https://www.sigmaaldrich.com/catalog/product/sial/331635?lang=en&region=IR) were purchased from Sigma Aldrich. Stock solutions of (1 mg.mL−1 Ni(II) and nitrate ions) were purchased from Merck, and standard solutions were prepared by appropriate dilution with Milli-Q water.

**1.2. Preparation of nanocomposites**

**1.2.1. Synthesis of MoS2 flower-like:**

The nanomaterial of 2D MoS2 flower-like was synthesized through the hydrothermal method. Firstly, (2 mmol) (NH4)6Mo7O24.4H2O and (4.5 mmol) H2NCSNH2 were dissolved in 75 mL deionized water and stirred for 10 min. Then, the solution was brought into a stainless-steel autoclave and maintained at 200 for 24 h. The MoS2 product was separated and washed with deionized water three times and dried in a vacuum at 50 overnight.

**1.2.2. Synthesis of γ-Fe2O3 Nanoparticles:**

According to previous reports, the synthesis of superparamagnetic γ-Fe2O3 nanoparticles was carried out via the co-precipitation method (Ostovar et al., 2018; Ostovar et al., 2020). First, (0.401 g) FeCl2.4H2O and (1.092 g) FeCl3.6H2O were dissolved in 2 and 4 mL of a (2 M) HCl solution, respectively, and stirred (800 rpm) at room temperature for 15 min. Then, a NH4OH solution (0.7 M, 50 mL) was slowly added to the mixture at room temperature that led to the color change of the solution from orange to dark brown. The obtained black precipitate was collected by an external magnet and was added to 1-2 mL of tetramethylammonium hydroxide surfactant. Subsequently, the obtained solid was washed several times with a mixture of H2O:EtOH (1:1) and dried at 100 overnight. Finally, the material was calcined at 300 for 3 h and synthesized superparamagnetic γ-Fe2O3 nanoparticles were obtained.

**1.3. Nanocomposite characterization.**

For powder X-ray diffraction patterns (XRD) measurement, a Bruker D8-advance X-ray diffract meter with Cu Kα radiation (λ = 0.154 nm) was used over the 2θ range of 10–80°.The Brunauer–Emmett–Teller (Bethi et al.) specific surface area and pore size measurements were accomplished by nitrogen adsorption at 77 K using a BETSORP Max surface area analyzer. In addition, structural and elemental compositions were characterized by the JEOL-SEM JSM-7800 LV scanning electron microscope (JEOL, Dearborn Rd, Peabody, USA) with an energy dispersive X-ray spectrometer (SEM-EDX). TEM images were acquired using a Zeiss EM 900 electron microscope. Thermogravimetric analysis (TGA) was performed on a Perkin-Elmer thermal analyzer by heating the sample up to 1000 at 20 min−1 under nitrogen atmosphere. In addition, to analyze the adsorbent materials, Fourier Transform Infra-Red (FT-IR) spectra were recorded by a Nicolet 6700 FT-IR spectrometer using KBr disc, and the spectra were recorded from 4000 to 450 cm−1 with a resolution of 4 cm−1. The magnetic properties of the samples were performed by using the vibrating sample magnetometer (VSM)-LAKESHORE (Model: 7404, Lake Shore Cryotronics, Westerville OH, USA) at room temperature. The ultraviolet-visible diffuse reflectance (UV-JASCO-570) spectra were obtained with the aid of a UV–Vis–NIR spectrophotometer (Lambda 950, Perkin Elmer) and the spectra were recorded at the wavelength range of 250–800 nm.

|  |  |
| --- | --- |
|  |  |
|  |  |
|  |  |

***Fig. S1:*** *BET measurements for A1) γ-Fe2O3 NPs, B1) MoS2, C1) MoS2/γ-Fe2O3(20%) nanocomposite, and Pore-size distributions (BJH, desorption branch) of A2) γ-Fe2O3 nanoparticles, B2) MoS2, and C2) MoS2/γ-Fe2O3(20%) nanocomposite*

|  |  |
| --- | --- |
| **A** | **B** |

***Fig. S2:*** *The morphology (A) MoS2/γ-Fe2O3(10%), and B) MoS2/γ-Fe2O3(30%) compositions*

|  |  |
| --- | --- |
| **A) Mo, S** | **Mo** |
| **S** | |
| **B) Mo, S, Fe, O** | **Mo** |
| **S** | **Fe** |
| **O** | |

***Figure S3****. EDX elemental mappings of A) MoS2 and B) MoS2/γ-Fe2O3(20%) nanocomposite*

|  |
| --- |
|  |
|  |

***Figure S4.*** *EDX spectra of A) MoS2, and B) MoS2/γ-Fe2O3(20%) nanocompsite.*

**Table S1.** Elemental distribution expressed in weight percentage (atomic percentage) in MoS2/γ-Fe2O3(20%) particles, as determined from SEMEDX analysis and values in atomic percentage as determined by ICP-MS analysis.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Catalyst** | **SEM/EDX (%Wt,%At)** | | | | **ICP-MS (%)** | | |
| **Mo** | **S** | **O** | **Fe** | **Fe** | **Mo** | **S** |
| **MoS2** | (68.26) ,(41.82) | (31.74) ,(58.18) | - | - | - |  |  |
| **MoS2/γ-Fe2O3(20%)** | (57.79) ,(28.24) | (34.57) ,(50.56) | (7.07) ,(20.71) | (0.57) ,(0.48) | 17.0 | 4.5 | 6.8 |

|  |
| --- |
|  |
|  |

***Fig. S5:*** *TGA (blue and green lines) and DTG (red lines) analyses of A) MoS2, and B) MoS2/γ-Fe2O3(20%) nanocomposite.*

|  |  |
| --- | --- |
|  |  |
|  |  |
|  | |

***Fig. S6:*** *FTIR spectra of A) γ-Fe2O3 nanoparticless, B) MoS2, C) MoS2/γ-Fe2O3(10%), D) MoS2/γ-Fe2O3(20%), and E) MoS2/γ-Fe2O3(30%) nanocomposite.*

|  |
| --- |
|  |
| **B)** |

***Fig. S7:*** *A)**Magnetic hysteresis loops of A1) γ-Fe2O3 and A2) MoS2/γ-Fe2O3(20%), B) superparamagnetic MoS2/γ-Fe2O3 nanocomposite collected by using an external magnet after the reaction*



***Fig. S8****: Point of zero charge (pHPZC) of the MoS2/γ-Fe2O3(20%) nanocomposite, determined by the pHdrift method (Matsumura et al., 2003)*

|  |  |
| --- | --- |
| **A** | **B** |
|  | |

***Fig. S9:*** *SEM image of the recovered A) the MoS2/γ-Fe2O3(20%) nanocomposite and B) the MoS2/γ-Fe2O3(30%) nanocomposite, and FTIR spectra of C) MoS2/γ-Fe2O3(20%), to remove Ni+2 ions after 6th cycle*

|  |  |
| --- | --- |
|  |  |
|  |  |
|  |  |

***Fig. S10****: Adsorption capacity and removal efficiency of ion for A) MoS2/γ-Fe2O3(10%), B) MoS2/γ-Fe2O3(20%), and C) MoS2/γ-Fe2O3(30%)* *([] = 0.1×10-4-0.5×10-4 M, [catalyst] = 0.005, 0.01 g, temperature = 25, 50 °C, pH = 7, time = 50min)*.

|  |  |
| --- | --- |
|  |  |
|  |  |
|  |  |

***Fig. S11****: Adsorption capacity and removal efficiency of Ni+2 ion for A) MoS2/γ-Fe2O3(10%), B) MoS2/γ-Fe2O3(20%), and C) MoS2/γ-Fe2O3(30%)* *([*Ni+2*] = 0.1-0.5 M, [catalyst] = 0.005, 0.01 g, temperature =25, 50 °C, pH = 8, time = 40 min)*.

**Table S2.** Empirical data for the catalytic performance of MoS2/γ-Fe2O3(10%) catalyst under different synthesis conditions (at different (a) Ni+2 and (b) concentrations)

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **(a)**  **Run** | **A**  **Ni Conc. (mol/L)** | **B**  **gcatal (g)** | **C**  **T (ºC)** | **Qe (mg/g)** | **Removal (%)** |
| 1 | 0.10 | 0.005 | 25.00 | 0.0603865 | 30 |
| 2 | 0.10 | 0.005 | 50.00 | 0.0743961 | 37 |
| 3 | 0.10 | 0.010 | 25.00 | 0.025 | 25 |
| 4 | 0.10 | 0.010 | 50.00 | 0.0333333 | 33 |
| 5 | 0.20 | 0.005 | 25.00 | 0.160261 | 40 |
| 6 | 0.20 | 0.005 | 50.00 | 0.194203 | 48 |
| 7 | 0.20 | 0.010 | 25.00 | 0.0644928 | 32 |
| 8 | 0.20 | 0.010 | 50.00 | 0.0886473 | 44 |
| 9 | 0.30 | 0.005 | 25.00 | 0.312261 | 52 |
| 10 | 0.30 | 0.005 | 50.00 | 0.355556 | 59 |
| 11 | 0.30 | 0.010 | 25.00 | 0.147585 | 49 |
| 12 | 0.30 | 0.010 | 50.00 | 0.1657 | 55 |
| 13 | 0.40 | 0.005 | 25.00 | 0.482261 | 60 |
| 14 | 0.40 | 0.005 | 50.00 | 0.560386 | 70 |
| 15 | 0.40 | 0.010 | 25.00 | 0.230797 | 57 |
| 16 | 0.40 | 0.010 | 50.00 | 0.247585 | 61 |
| 17 | 0.50 | 0.005 | 25.00 | 0.742261 | 74 |
| 18 | 0.50 | 0.005 | 50.00 | 0.803865 | 80 |
| 19 | 0.50 | 0.010 | 25.00 | 0.343961 | 68 |
| 20 | 0.50 | 0.010 | 50.00 | 0.38744 | 77 |

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **(b)**  **Run** | **A**  **Conc. (×10-4) (mol/L)** | **B**  **gcatal (g)** | **C**  **T (ºC)** | **Qe (mg/g)** | **Removal (%)** |
| 1 | 0.10 | 0.005 | 25.00 | 6.5934E-006 | 32 |
| 2 | 0.10 | 0.005 | 50.00 | 7.44868E-006 | 37 |
| 3 | 0.10 | 0.010 | 25.00 | 2.82629E-006 | 28 |
| 4 | 0.10 | 0.010 | 50.00 | 3.4143E-006 | 34 |
| 5 | 0.20 | 0.005 | 25.00 | 1.56885E-005 | 39 |
| 6 | 0.20 | 0.005 | 50.00 | 1.84682E-005 | 46 |
| 7 | 0.20 | 0.010 | 25.00 | 7.09588E-006 | 35 |
| 8 | 0.20 | 0.010 | 50.00 | 8.46433E-006 | 42 |
| 9 | 0.30 | 0.005 | 25.00 | 2.88462E-005 | 48 |
| 10 | 0.30 | 0.005 | 50.00 | 3.33365E-005 | 55 |
| 11 | 0.30 | 0.010 | 25.00 | 1.36747E-005 | 45 |
| 12 | 0.30 | 0.010 | 50.00 | 1.54281E-005 | 51 |
| 13 | 0.40 | 0.005 | 25.00 | 4.58527E-005 | 57 |
| 14 | 0.40 | 0.005 | 50.00 | 5.05568E-005 | 63 |
| 15 | 0.40 | 0.010 | 25.00 | 2.08951E-005 | 52 |
| 16 | 0.40 | 0.010 | 50.00 | 2.36747E-005 | 59 |
| 17 | 0.50 | 0.005 | 25.00 | 6.56389E-005 | 65 |
| 18 | 0.50 | 0.005 | 50.00 | 7.0343E-005 | 70 |
| 19 | 0.50 | 0.010 | 25.00 | 3.11089E-005 | 62 |
| 20 | 0.50 | 0.010 | 50.00 | 3.41024E-005 | 68 |

**Table S3**. Empirical data for the catalytic performance of MoS2/γ-Fe2O3(20%) catalyst under different synthesis conditions (at different (a) Ni+2 and (b) concentrations).

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **(a)**  **Run** | **A**  **Ni Conc. (mol/L)** | **B**  **gcatal (g)** | **C**  **T (ºC)** | **Qe (mg/g)** | **Removal (%)** |
| 1 | 0.10 | 0.005 | 25.00 | 0.13236715 | 66 |
| 2 | 0.10 | 0.005 | 50.00 | 0.14057971 | 70 |
| 3 | 0.10 | 0.010 | 25.00 | 0.060024155 | 60 |
| 4 | 0.10 | 0.010 | 50.00 | 0.068357488 | 68 |
| 5 | 0.20 | 0.005 | 25.00 | 0.28826087 | 72 |
| 6 | 0.20 | 0.005 | 50.00 | 0.312560386 | 78 |
| 7 | 0.20 | 0.010 | 25.00 | 0.136956522 | 68 |
| 8 | 0.20 | 0.010 | 50.00 | 0.150241546 | 75 |
| 9 | 0.30 | 0.005 | 25.00 | 0.48826087 | 81 |
| 10 | 0.30 | 0.005 | 50.00 | 0.524637681 | 87 |
| 11 | 0.30 | 0.010 | 25.00 | 0.228502415 | 76 |
| 12 | 0.30 | 0.010 | 50.00 | 0.252657005 | 84 |
| 13 | 0.40 | 0.005 | 25.00 | 0.71426087 | 89 |
| 14 | 0.40 | 0.005 | 50.00 | 0.753623188 | 94 |
| 15 | 0.40 | 0.010 | 25.00 | 0.34178744 | 85 |
| 16 | 0.40 | 0.010 | 50.00 | 0.362318841 | 90 |
| 17 | 0.50 | 0.005 | 25.00 | 0.92626087 | 92 |
| 18 | 0.50 | 0.005 | 50.00 | 0.970531401 | 97 |
| 19 | 0.50 | 0.010 | 25.00 | 0.446618357 | 89 |
| 20 | 0.50 | 0.010 | 50.00 | 0.468357488 | 93 |

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **(b)**  **Run** | **A**  **Conc. (×10-4) (mol/L)** | **B**  **gcatal (g)** | **C**  **T (ºC)** | **Qe (mg/g)** | **Removal (%)** |
| 1 | 0.10 | 0.005 | 25.00 | 1.19389E-05 | 59 |
| 2 | 0.10 | 0.005 | 50.00 | 1.3008E-05 | 65 |
| 3 | 0.10 | 0.010 | 25.00 | 5.64873E-06 | 56 |
| 4 | 0.10 | 0.010 | 50.00 | 6.18329E-06 | 61 |
| 5 | 0.20 | 0.005 | 25.00 | 2.65934E-05 | 66 |
| 6 | 0.20 | 0.005 | 50.00 | 2.89454E-05 | 72 |
| 7 | 0.20 | 0.010 | 25.00 | 1.27621E-05 | 63 |
| 8 | 0.20 | 0.010 | 50.00 | 1.39382E-05 | 69 |
| 9 | 0.30 | 0.005 | 25.00 | 4.33861E-05 | 72 |
| 10 | 0.30 | 0.005 | 50.00 | 4.65934E-05 | 77 |
| 11 | 0.30 | 0.010 | 25.00 | 2.06239E-05 | 68 |
| 12 | 0.30 | 0.010 | 50.00 | 2.26552E-05 | 75 |
| 13 | 0.40 | 0.005 | 25.00 | 6.57381E-05 | 82 |
| 14 | 0.40 | 0.005 | 50.00 | 7.08698E-05 | 88 |
| 15 | 0.40 | 0.010 | 25.00 | 3.07308E-05 | 76 |
| 16 | 0.40 | 0.010 | 50.00 | 3.37243E-05 | 84 |
| 17 | 0.50 | 0.005 | 25.00 | 8.95869E-05 | 89 |
| 18 | 0.50 | 0.005 | 50.00 | 9.40771E-05 | 94 |
| 19 | 0.50 | 0.010 | 25.00 | 4.31898E-05 | 86 |
| 20 | 0.50 | 0.010 | 50.00 | 4.50073E-05 | 90 |

**Table S4**. Empirical data for the catalytic performance of MoS2/γ-Fe2O3(30%) catalyst under different synthesis conditions (at different (a) Ni+2 and (b) concentrations).

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **(a)**  **Run** | **A**  **Ni Conc. (mol/L)** | **B**  **gcatal (g)** | **C**  **T (ºC)** | **Qe (mg/g)** | **Removal (%)** |
| 1 | 0.10 | 0.005 | 25.00 | 0.134299517 | 67 |
| 2 | 0.10 | 0.005 | 50.00 | 0.142753623 | 71 |
| 3 | 0.10 | 0.010 | 25.00 | 0.062439614 | 62 |
| 4 | 0.10 | 0.010 | 50.00 | 0.069565217 | 69 |
| 5 | 0.20 | 0.005 | 25.00 | 0.29826087 | 74 |
| 6 | 0.20 | 0.005 | 50.00 | 0.322222222 | 80 |
| 7 | 0.20 | 0.010 | 25.00 | 0.139371981 | 69 |
| 8 | 0.20 | 0.010 | 50.00 | 0.152657005 | 76 |
| 9 | 0.30 | 0.005 | 25.00 | 0.50226087 | 83 |
| 10 | 0.30 | 0.005 | 50.00 | 0.543961353 | 90 |
| 11 | 0.30 | 0.010 | 25.00 | 0.234541063 | 78 |
| 12 | 0.30 | 0.010 | 50.00 | 0.262318841 | 87 |
| 13 | 0.40 | 0.005 | 25.00 | 0.73226087 | 91 |
| 14 | 0.40 | 0.005 | 50.00 | 0.760869565 | 95 |
| 15 | 0.40 | 0.010 | 25.00 | 0.350241546 | 87 |
| 16 | 0.40 | 0.010 | 50.00 | 0.3647343 | 91 |
| 17 | 0.50 | 0.005 | 25.00 | 0.93226087 | 93 |
| 18 | 0.50 | 0.005 | 50.00 | 0.980193237 | 98 |
| 19 | 0.50 | 0.010 | 25.00 | 0.456280193 | 91 |
| 20 | 0.50 | 0.010 | 50.00 | 0.473188406 | 94 |

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **(b)**  **Run** | **A**  **Conc. (×10-4) (mol/L)** | **B**  **gcatal (g)** | **C**  **T (ºC)** | **Qe (mg/g)** | **Removal (%)** |
| 1 | 0.10 | 0.005 | 25.00 | 1.25911E-05 | 62 |
| 2 | 0.10 | 0.005 | 50.00 | 1.34357E-05 | 67 |
| 3 | 0.10 | 0.010 | 25.00 | 5.96947E-06 | 59 |
| 4 | 0.10 | 0.010 | 50.00 | 6.54678E-06 | 65 |
| 5 | 0.20 | 0.005 | 25.00 | 2.70424E-05 | 67 |
| 6 | 0.20 | 0.005 | 50.00 | 2.94158E-05 | 73 |
| 7 | 0.20 | 0.010 | 25.00 | 1.30829E-05 | 65 |
| 8 | 0.20 | 0.010 | 50.00 | 1.42589E-05 | 71 |
| 9 | 0.30 | 0.005 | 25.00 | 4.44552E-05 | 74 |
| 10 | 0.30 | 0.005 | 50.00 | 4.85178E-05 | 80 |
| 11 | 0.30 | 0.010 | 25.00 | 2.13723E-05 | 71 |
| 12 | 0.30 | 0.010 | 50.00 | 2.31898E-05 | 77 |
| 13 | 0.40 | 0.005 | 25.00 | 6.68072E-05 | 83 |
| 14 | 0.40 | 0.005 | 50.00 | 7.12975E-05 | 89 |
| 15 | 0.40 | 0.010 | 25.00 | 3.17999E-05 | 79 |
| 16 | 0.40 | 0.010 | 50.00 | 3.4152E-05 | 85 |
| 17 | 0.50 | 0.005 | 25.00 | 9.00145E-05 | 90 |
| 18 | 0.50 | 0.005 | 50.00 | 9.51463E-05 | 95 |
| 19 | 0.50 | 0.010 | 25.00 | 4.40451E-05 | 88 |
| 20 | 0.50 | 0.010 | 50.00 | 4.66109E-05 | 93 |

**Table S5.** ANOVA table for Qe of MoS2/γ-Fe2O3(10%) catalyst with different Ni+2 concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 0.98 | 5 | 0.20 | 639.91 | < 0.0001 | significant |
| A- Ni+2 Conc. | 0.67 | 1 | 0.67 | 2191.50 | < 0.0001 |  |
| B-Cat. | 0.20 | 1 | 0.20 | 660.22 | < 0.0001 |  |
| C-T | 5.843E-003 | 1 | 5.843E-003 | 19.07 | 0.0006 |  |
| AB | 0.085 | 1 | 0.085 | 276.12 | < 0.0001 |  |
| A^2 | 0.016 | 1 | 0.016 | 52.65 | < 0.0001 |  |
| Residual | 4.289E-003 | 14 | 3.064E-004 |  |  |  |
| Cor Total | 0.98 | 19 |  |  |  |  |
| R-Squared=0.9956  Adj R-Squared= 0.9941  Pred R-Squared= 0.9902  Adeq Precision= 78.601  Std. Dev.= 0.018 | | | | | | |

**Table S6.** ANOVA table for removal percent of MoS2/γ-Fe2O3(10%) catalyst with different Ni+2 concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 5082.10 | 3 | 1694.03 | 604.34 | < 0.0001 | significant |
| A- Ni+2 Conc. | 4665.60 | 1 | 4665.60 | 1664.43 | < 0.0001 |  |
| B-Cat. | 120.05 | 1 | 120.05 | 42.83 | < 0.0001 |  |
| C-T | 296.45 | 1 | 296.45 | 105.76 | < 0.0001 |  |
| Residual | 44.85 | 16 | 2.80 |  |  |  |
| Cor Total | 5126.95 | 19 |  |  |  |  |
| R-Squared=0.9913  Adj R-Squared=0.9896  Pred R-Squared=0.9870  Adeq Precision=74.524  Std. Dev.= 1.67 | | | | | | |

**Table S7.** ANOVA table for Qe of MoS2/γ-Fe2O3(10%) catalyst with different concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 7.553E-009 | 6 | 1.259E-009 | 1278.67 | < 0.0001 | significant |
| A- Conc. (10-4) | 5.132E-009 | 1 | 5.132E-009 | 5213.08 | < 0.0001 |  |
| B-Cat. | 1.658E-009 | 1 | 1.658E-009 | 1683.96 | < 0.0001 |  |
| C-T | 3.649E-011 | 1 | 3.649E-011 | 37.07 | < 0.0001 |  |
| AB | 6.335E-010 | 1 | 6.335E-010 | 643.46 | < 0.0001 |  |
| AC | 6.276E-012 | 1 | 6.276E-012 | 6.37 | 0.0254 |  |
| A^2 | 8.671E-011 | 1 | 8.671E-011 | 88.08 | < 0.0001 |  |
| Residual | 1.280E-011 | 13 | 9.845E-013 |  |  |  |
| Cor Total | 7.566E-009 | 19 |  |  |  |  |
| R-Squared= 0.9983  Adj R-Squared= 0.9975  Pred R-Squared= 0.9953  Adeq Precision= 112.810  Std. Dev.= 9.922E-007 | | | | | | |

**Table S8.** ANOVA table for removal percent of MoS2/γ-Fe2O3(10%) catalyst with different concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 3096.23 | 3 | 1032.08 | 2511.51 | < 0.0001 | significant |
| A- Conc. (10-4) | 2839.23 | 1 | 2839.23 | 6909.14 | < 0.0001 |  |
| B-Cat. | 64.80 | 1 | 64.80 | 157.69 | < 0.0001 |  |
| C-T | 192.20 | 1 | 192.20 | 467.71 | < 0.0001 |  |
| Residual | 6.57 | 16 | 0.41 |  |  |  |
| Cor Total | 3102.80 | 19 |  |  |  |  |
| R-Squared= 0.9979  Adj R-Squared= 0.9975  Pred R-Squared= 0.9966  Adeq Precision= 151.735  Std. Dev.= 0.64 | | | | | | |

**Table S9.** ANOVA table for Qe of MoS2/γ-Fe2O3(20%) catalyst with different Ni+2 concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 1.42 | 5 | 0.28 | 2386.85 | < 0.0001 | significant |
| A- Ni+2 Conc. | 0.93 | 1 | 0.93 | 7807.64 | < 0.0001 |  |
| B-Cat. | 0.37 | 1 | 0.37 | 3135.31 | < 0.0001 |  |
| C-T | 2.894E-003 | 1 | 2.894E-003 | 24.25 | 0.0002 |  |
| AB | 0.11 | 1 | 0.11 | 945.69 | < 0.0001 |  |
| A^2 | 2.552E-003 | 1 | 2.552E-003 | 21.39 | 0.0004 |  |
| Residual | 1.671E-003 | 14 | 1.193E-004 |  |  |  |
| Cor Total | 1.43 | 19 |  |  |  |  |
| R-Squared=0.9988  Adj R-Squared=0.9984  Pred R-Squared=0.9974  Adeq Precision=151.770  Std. Dev.= 0.011 | | | | | | |

**Table S10.** ANOVA table for removal percent of MoS2/γ-Fe2O3(20%) catalyst with different Ni+2 concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 2210.87 | 4 | 552.72 | 355.39 | < 0.0001 | significant |
| A- Ni+2 Conc. | 1946.03 | 1 | 1946.03 | 1251.27 | < 0.0001 |  |
| B-Cat. | 72.20 | 1 | 72.20 | 46.42 | < 0.0001 |  |
| C-T | 168.20 | 1 | 168.20 | 108.15 | < 0.0001 |  |
| A^2 | 24.45 | 1 | 24.45 | 15.72 | 0.0012 |  |
| Residual | 23.33 | 15 | 1.56 |  |  |  |
| Cor Total | 2234.20 | 19 |  |  |  |  |
| R-Squared= 0.9896  Adj R-Squared= 0.9868  Pred R-Squared= 0.9813  Adeq Precision= 60.140  Std. Dev.= 1.25 | | | | | | |

**Table S11.** ANOVA table for Qe of MoS2/γ-Fe2O3(20%) catalyst with different concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 1.315E-008 | 5 | 2.630E-009 | 1605.22 | < 0.0001 | significant |
| A- Conc. (10-4) | 8.673E-009 | 1 | 8.673E-009 | 5293.23 | < 0.0001 |  |
| B-Cat. | 3.284E-009 | 1 | 3.284E-009 | 2004.22 | < 0.0001 |  |
| C-T | 3.076E-011 | 1 | 3.076E-011 | 18.77 | 0.0007 |  |
| AB | 1.082E-009 | 1 | 1.082E-009 | 660.25 | < 0.0001 |  |
| A^2 | 8.127E-011 | 1 | 8.127E-011 | 49.60 | < 0.0001 |  |
| Residual | 2.294E-011 | 14 | 1.638E-012 |  |  |  |
| Cor Total | 1.317E-008 | 19 |  |  |  |  |
| R-Squared= 0.9983  Adj R-Squared= 0.9976  Pred R-Squared= 0.9961  Adeq Precision= 124.101  Std. Dev.= 1.280E-006 | | | | | | |

**Table S12.** ANOVA table for removal percent of MoS2/γ-Fe2O3(20%) catalyst with different concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 2423.40 | 3 | 807.80 | 603.96 | < 0.0001 | significant |
| A- Conc. (10-4) | 2190.40 | 1 | 2190.40 | 1637.68 | < 0.0001 |  |
| B-Cat. | 64.80 | 1 | 64.80 | 48.45 | < 0.0001 |  |
| C-T | 168.20 | 1 | 168.20 | 125.76 | < 0.0001 |  |
| Residual | 21.40 | 16 | 1.34 |  |  |  |
| Cor Total | 2444.80 | 19 |  |  |  |  |
| R-Squared= 0.9912  Adj R-Squared= 0.9896  Pred R-Squared= 0.9871  Adeq Precision= 75.405  Std. Dev.= 1.16 | | | | | | |

**Table S13.** ANOVA table for Qe of MoS2/γ-Fe2O3(30%) catalyst with different Ni+2 concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 1.45 | 5 | 0.29 | 2517.80 | < 0.0001 | significant |
| A- Ni+2 Conc. | 0.95 | 1 | 0.95 | 8218.50 | < 0.0001 |  |
| B-Cat. | 0.39 | 1 | 0.39 | 3355.94 | < 0.0001 |  |
| C-T | 2.651E-003 | 1 | 2.651E-003 | 22.95 | 0.0003 |  |
| AB | 0.11 | 1 | 0.11 | 978.13 | < 0.0001 |  |
| A^2 | 1.556E-003 | 1 | 1.556E-003 | 13.47 | 0.0025 |  |
| Residual | 1.617E-003 | 14 | 1.155E-004 |  |  |  |
| Cor Total | 1.46 | 19 |  |  |  |  |
| R-Squared=0.9989  Adj R-Squared=0.9985  Pred R-Squared=0.9975  Adeq Precision=155.892  Std. Dev.= 0.011 | | | | | | |

**Table S14.** ANOVA table for removal percent of MoS2/γ-Fe2O3(30%) catalyst with different Ni+2 concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 2217.90 | 4 | 554.47 | 257.50 | < 0.0001 | significant |
| A- Ni+2 Conc. | 1946.02 | 1 | 1946.02 | 903.73 | < 0.0001 |  |
| B-Cat. | 72.20 | 1 | 72.20 | 33.53 | < 0.0001 |  |
| C-T | 156.80 | 1 | 156.80 | 72.82 | < 0.0001 |  |
| A^2 | 42.88 | 1 | 42.88 | 19.91 | 0.0005 |  |
| Residual | 32.30 | 15 | 2.15 |  |  |  |
| Cor Total | 2250.20 | 19 |  |  |  |  |
| R-Squared= 0.9856  Adj R-Squared= 0.9818  Pred R-Squared= 0.9750  Adeq Precision= 50.837  Std. Dev.= 1.47 | | | | | | |

**Table S15.** ANOVA table for Qe of MoS2/γ-Fe2O3(30%) catalyst with different concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 1.334E-008 | 7 | 1.905E-009 | 2688.93 | < 0.0001 | significant |
| A- Conc. (10-4) | 8.845E-009 | 1 | 8.845E-009 | 12484.18 | < 0.0001 |  |
| B-Cat. | 3.320E-009 | 1 | 3.320E-009 | 4686.58 | < 0.0001 |  |
| C-T | 3.224E-011 | 1 | 3.224E-011 | 45.50 | < 0.0001 |  |
| AB | 1.051E-009 | 1 | 1.051E-009 | 1483.29 | < 0.0001 |  |
| AC | 6.276E-012 | 1 | 6.276E-012 | 8.86 | 0.0116 |  |
| BC | 3.540E-012 | 1 | 3.540E-012 | 5.00 | 0.0452 |  |
| A^2 | 7.730E-011 | 1 | 7.730E-011 | 109.10 | < 0.0001 |  |
| Residual | 8.502E-012 | 12 | 7.085E-013 |  |  |  |
| Cor Total | 1.334E-008 | 19 |  |  |  |  |
| R-Squared= 0.9994  Adj R-Squared= 0.9990  Pred R-Squared=0.9977  Adeq Precision=164.910  Std. Dev.= 8.417E-007 | | | | | | |

**Table S16.** ANOVA table for removal percent of MoS2/γ-Fe2O3(30%) catalyst with different concentration.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 2252.44 | 4 | 563.11 | 1383.08 | < 0.0001 | significant |
| A- Conc. (10-4) | 2044.90 | 1 | 2044.90 | 5022.56 | < 0.0001 |  |
| B-Cat. | 36.45 | 1 | 36.45 | 89.53 | < 0.0001 |  |
| C-T | 162.45 | 1 | 162.45 | 399.00 | < 0.0001 |  |
| A^2 | 8.64 | 1 | 8.64 | 21.23 | 0.0003 |  |
| Residual | 6.11 | 15 | 0.41 |  |  |  |
| Cor Total | 2258.55 | 19 |  |  |  |  |
| R-Squared= 0.9973  Adj R-Squared= 0.9966  Pred R-Squared= 0.9954  Adeq Precision= 115.973  Std. Dev.= 0.64 | | | | | | |

|  |  |
| --- | --- |
|  |  |
|  |  |
|  |  |

***Fig. S12:*** *The diagnostic plots for Qe (1) and removal percent (2) responses of MoS2/γ-Fe2O3(10%) catalyst at different Ni+2 concentration: normal probability plot of residuals (a), residuals versus predicted (b), and predicted versus actual plots (c).*

|  |  |
| --- | --- |
|  |  |
|  |  |
|  |  |

***Fig. S13:*** *The diagnostic plots for Qe (1) and removal percent (2) responses of MoS2/γ-Fe2O3(10%) catalyst at different concentration: normal probability plot of residuals (a), residuals versus predicted (b), and predicted versus actual plots (c).*

|  |  |
| --- | --- |
| **a1** | **a2** |
| **b1** | **b2** |
| **c1** | **c2** |

***Fig. S14:*** *The diagnostic plots for Qe (1) and removal percent (2) responses of MoS2/γ-Fe2O3(20%) catalyst at different Ni+2 concentration: normal probability plot of residuals (a), residuals versus predicted (b), and predicted versus actual plots (c).*

|  |  |
| --- | --- |
| **a1** | **a2** |
| **b1** | **b2** |
| **c1** | **c2** |

***Fig. S15:*** *The diagnostic plots for Qe (1) and removal percent (2) responses of MoS2/γ-Fe2O3(20%) catalyst at different concentration: normal probability plot of residuals (a), residuals versus predicted (b), and predicted versus actual plots (c).*

|  |  |
| --- | --- |
| **a1** | **a2** |
| **b1** | **b2** |
| **c1** | **c2** |

***Fig. S16:*** *The diagnostic plots for Qe (1) and removal percent (2) responses of MoS2/γ-Fe2O3(30%) catalyst at different Ni+2 concentration: normal probability plot of residuals (a), residuals versus predicted (b), and predicted versus actual plots (c).*

|  |  |
| --- | --- |
| **a1** | **a2** |
| **b1** | **b2** |
| **c1** | **c2** |

***Fig. S17:*** *The diagnostic plots for Qe (1) and removal percent (2) responses of MoS2/γ-Fe2O3(30%) catalyst at different concentration: normal probability plot of residuals (a), residuals versus predicted (b), and predicted versus actual plots (c).*

**Table S17.** ANOVA table for Ni+2 species and Qe response.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 1.01 | 5 | 0.20 | 95.72 | < 0.0001 | significant |
| A-Fe2O3 | 0.031 | 1 | 0.031 | 14.82 | 0.0063 |  |
| B- Ni+2 Conc. | 0.96 | 1 | 0.96 | 453.78 | < 0.0001 |  |
| AB | 2.914E-003 | 1 | 2.914E-003 | 1.38 | 0.2783 |  |
| A^2 | 0.018 | 1 | 0.018 | 8.59 | 0.0220 |  |
| B^2 | 1.698E-003 | 1 | 1.698E-003 | 0.80 | 0.3994 |  |
| Residual | 0.015 | 7 | 2.110E-003 |  |  |  |
| Lack of Fit | 3.884E-003 | 3 | 1.295E-003 | 0.48 | 0.7161 | insignificant |
| Pure Error | 0.011 | 4 | 2.722E-003 |  |  |  |
| Cor Total | 1.02 | 12 |  |  |  |  |
| R-Squared= 0.9856  Adj R-Squared= 0.9753  Pred R-Squared= 0.9515  Adeq Precision= 30.228  Std. Dev.=0.046 | | | | | | |

**Table S18.** ANOVA table for Ni+2 species and removal percent response.

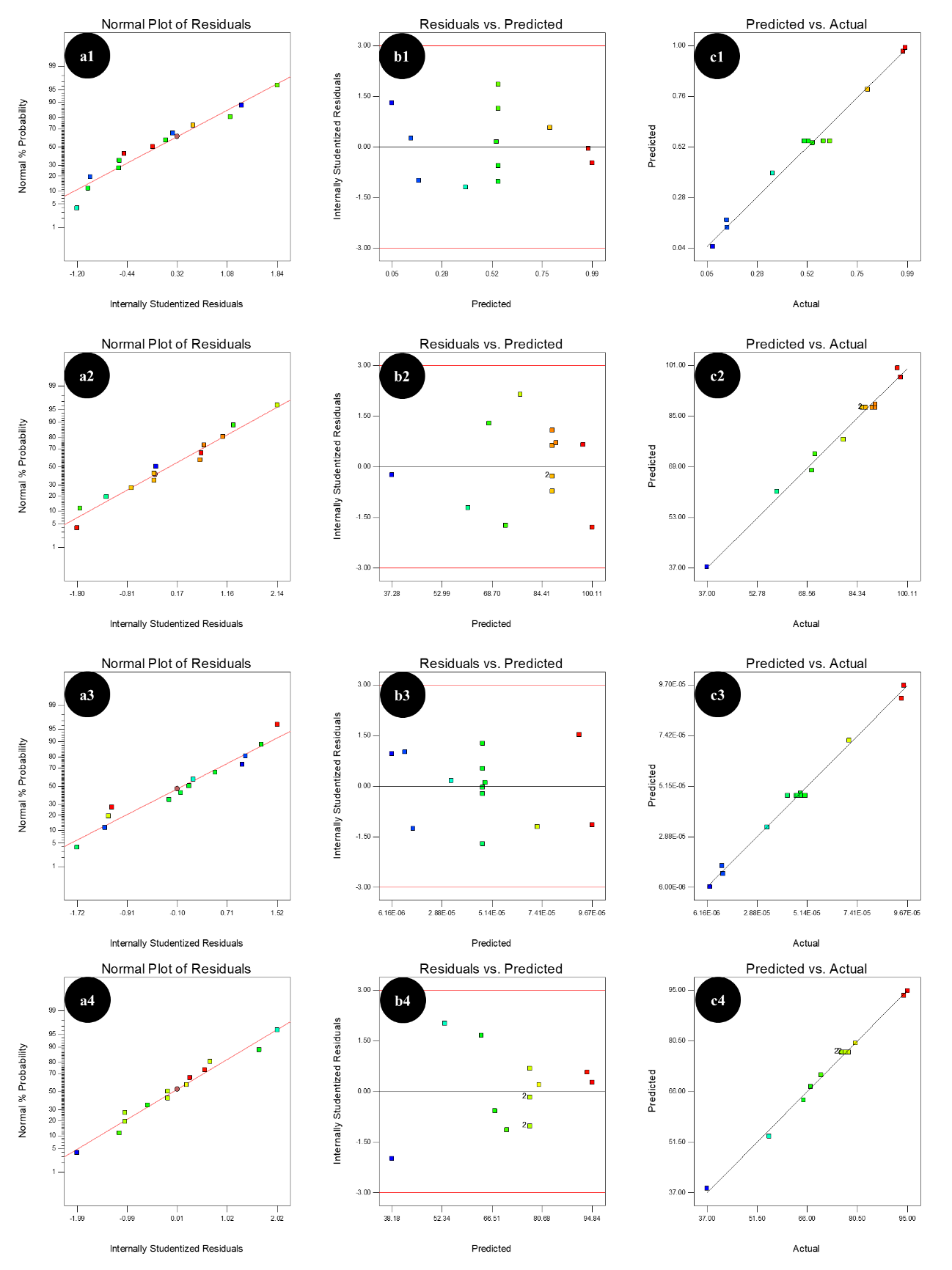
|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 3457.29 | 5 | 691.46 | 116.26 | < 0.0001 | significant |
| A-Fe2O3 | 1148.17 | 1 | 1148.17 | 193.05 | < 0.0001 |  |
| B- Ni+2 Conc. | 1568.17 | 1 | 1568.17 | 263.67 | < 0.0001 |  |
| AB | 64.00 | 1 | 64.00 | 10.76 | 0.0135 |  |
| A^2 | 443.53 | 1 | 443.53 | 74.58 | < 0.0001 |  |
| B^2 | 37.25 | 1 | 37.25 | 6.26 | 0.0408 |  |
| Residual | 41.63 | 7 | 5.95 |  |  |  |
| Lack of Fit | 30.83 | 3 | 10.28 | 3.81 | 0.1147 | insignificant |
| Pure Error | 10.80 | 4 | 2.70 |  |  |  |
| Cor Total | 3498.92 | 12 |  |  |  |  |
| R-Squared= 0.9881  Adj R-Squared= 0.9796  Pred R-Squared= 0.9069  Adeq Precision= 37.928  Std. Dev.=2.44 | | | | | | |

**Table S19.** ANOVA table for species and Qe response.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 9.089E-009 | 5 | 1.818E-009 | 208.40 | < 0.0001 | significant |
| A-Fe2O3 | 3.522E-010 | 1 | 3.522E-010 | 40.38 | 0.0004 |  |
| B-Conc. \*10-4 | 8.488E-009 | 1 | 8.488E-009 | 973.16 | < 0.0001 |  |
| AB | 8.851E-011 | 1 | 8.851E-011 | 10.15 | 0.0154 |  |
| A^2 | 1.183E-010 | 1 | 1.183E-010 | 13.56 | 0.0078 |  |
| B^2 | 1.018E-010 | 1 | 1.018E-010 | 11.67 | 0.0112 |  |
| Residual | 6.106E-011 | 7 | 8.722E-012 |  |  |  |
| Lack of Fit | 2.606E-011 | 3 | 8.685E-012 | 0.99 | 0.4815 | insignificant |
| Pure Error | 3.500E-011 | 4 | 8.750E-012 |  |  |  |
| Cor Total | 9.150E-009 | 12 |  |  |  |  |
| R-Squared= 0.9933  Adj R-Squared= 0.9886  Pred R-Squared= 0.9656  Adeq Precision= 45.130  Std. Dev.= 2.953E-006 | | | | | | |

**Table S20.** ANOVA table for species and removal percent response.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Source** | **Sum of squares** | **df** | **Mean square** | **F value** | **p-valueProb>F** |  |
| Model | 2766.06 | 5 | 553.21 | 332.83 | < 0.0001 | significant |
| A-Fe2O3 | 1066.67 | 1 | 1066.67 | 641.74 | < 0.0001 |  |
| B-Conc. \*10-4 | 1350.00 | 1 | 1350.00 | 812.20 | < 0.0001 |  |
| AB | 6.25 | 1 | 6.25 | 3.76 | 0.0937 |  |
| A^2 | 317.64 | 1 | 317.64 | 191.10 | < 0.0001 |  |
| B^2 | 4.50 | 1 | 4.50 | 2.70 | 0.1440 |  |
| Residual | 11.64 | 7 | 1.66 |  |  |  |
| Lack of Fit | 8.84 | 3 | 2.95 | 4.21 | 0.0994 | insignificant |
| Pure Error | 2.80 | 4 | 0.70 |  |  |  |
| Cor Total | 2777.69 | 12 |  |  |  |  |
| R-Squared= 0.9958  Adj R-Squared= 0.9928  Pred R-Squared= 0.9734  Adeq Precision= 64.698  Std. Dev.=1.29 | | | | | | |



***Fig. S18:*** *The diagnostic plots for Qe (1 and 3) and removal percent (2 and 4) responses for both species of Ni+2 (1 and 2) and (3 and 4): normal probability plot of residuals (a), residuals versus predicted (b) and predicted versus actual plots (c).*

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  | ***Table S21: Comparison of MoS2-based catalysts for the adsorption catalytic studies.*** | | | |
| **Catalyst** | | **Operation Conditions** | **Operation Conditions** | **[Ref]** | |
| **MoS2–rGO** | | [**Ni+2**] = 15 μg.L−1  volume of solution = 20 mL, pH = 6.8, eluent; 2 mL of 2 mol.L−1 HNO3, adsorbent amount 25 mg,  Qe(Ni+2) = 294 mg.g−1 | [Pb2+] = 20 μg.L−1, volume of solution = 20 mL, pH = 6.8, eluent; 2 mL of 2 mol.L−1 HNO3, adsorbent amount 25 mg, Qe(Pb2+) = 322 mg.g−1 | (Aghagoli et al., 2017) | |
| **MoS2/SH-MWCNT** | | Co = 100 mg mL−1; time = 60 min; and adsorbent dosage = 2 mg mL−1, Qe(Cd+2) = 66.6 mg.g−1 | Co = 100 mg mL−1; time = 60 min; and adsorbent dosage = 2 mg mL−1, Qe(Pb2+) = 90.0 mg g−1 | (Gusain et al., 2019) | |
| **MoS2/γ-Fe2O3 (10%)** | | [**Ni+2**] = 0.5 M, [catalyst]=0.005 g, temperature=50 °C, pH=8, time=40 min, Qe(Ni+2) = 0.80 mg.g−1, Removal%( Ni+2) =80.38% | [] = 0.5×10-4 M, [catalyst]=0.005 g, temperature=50 °C, pH=7, time=50 min,Qe()= 7.03×10-5 mg.g−1, Removal%() = 70.34% | **This work** | |
| **MoS2/γ-Fe2O3 (20%)** | | [Ni+2] =0.5 M, [catalyst]=0.005 g, temperature=50 °C, pH=8, time=40 min, Qe (Ni+2) = 0.97 mg.g−1, Removal% (Ni+2) =97.05% | []= 0.5×10-4 M, [catalyst]=0.005 g, temperature=50 °C, pH=7, time=50 min, Qe ()= 9.41×10-5 mg.g−1, Removal% () = 94.07% | **This work** | |
| **MoS2/γ-Fe2O3 (30%)** | | [Ni+2] =0.5 M, [catalyst]=0.005 g, temperature=50 °C, pH=8, time=40 min, Qe(Ni+2) =0.98 mg.g−1, Removal%(Ni+2) =98.01% | []= 0.5×10-4 M, [catalyst]=0.005 g, temperature=50 °C, pH=7, time=50 min, Qe() = 9.51×10-5 mg.g−1, Removal%() = 95.14% | **This work** | |

**Reference:**

Aghagoli, M. J., and Shemirani, F. 2017. Hybrid nanosheets composed of molybdenum disulfide and reduced graphene oxide for enhanced solid phase extraction of Pb (II) and Ni (II). Mikrochim. Acta, 184, 237-244.

Bethi, B., Sonawane, S. H., Bhanvase, B. A., and Gumfekar, S. P. 2016. Nanomaterials-based advanced oxidation processes for wastewater treatment: A review. Chem. Eng. Process.: Process Intensif., 109, 178-189.

Gusain, R., Kumar, N., Fosso-Kankeu, E., and Ray, S. S. 2019. Efficient removal of Pb (II) and Cd (II) from industrial mine water by a hierarchical MoS2/SH-MWCNT nanocomposite. ACS omega, 4(9), 13922-13935.

Matsumura, Y., Yoshikata, K., Kunisaki, S.-i., and Tsuchido, T. 2003. Mode of bactericidal action of silver zeolite and its comparison with that of silver nitrate. Appl. Environ. Microbiol., 69(7), 4278-4281.

Ostovar, S., Prinsen, P., Yepez, A., Shaterian, H. R., and Luque, R. 2018. Catalytic Versatility of Novel Sulfonamide Functionalized Magnetic Composites. ACS Sustain. Chem. Eng., 6(4), 4586-4593.

Ostovar, S., Rezvani, A., Luque, R., and Carrillo-Carrión, C. 2020. Core-shell iron oxide@ cathecol-polymer@ palladium/copper nanocomposites as efficient and sustainable catalysts in cross-coupling reactions. J. Mol. Catal., 493, 111042.